

# Diode Laser Measurements of HF Concentrations From Heptane/Air Pan Fires Extinguished by FE-36 and FE-36 Plus APP

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# **Army Research Laboratory**

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## **Diode Laser Measurements of HF Concentrations From Heptane/Air Pan Fires Extinguished by FE-36 and FE-36 Plus APP**

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## Abstract

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Tunable diode laser absorption spectroscopy (TDLAS) is used to measure the time evolution of hydrogen fluoride (HF) concentrations produced from a series of enclosed heptane/air pan fires extinguished by FE-36 or FE-36 plus ammonium polyphosphate (APP). Recent advances in room-temperature fiber-coupled near-infrared (NIR) diode lasers provide isolation of the laser and signal-processing electronics from the hostile sampling site. For the fires studied, the change in HF gas concentration with time is dependent upon the fire-fighting chemical used to extinguish the fire. The presence of APP is observed to accelerate the dissipation of HF from the fire enclosure. Visible attenuation spectroscopy is also used to measure the amount of light attenuation (obscuration) that occurs as a hand-held fire extinguisher containing powder fire-fighting agent is released in the crew space of a M1-Abrams land combat vehicle. Obscuration tests demonstrate that release of APP from extinguishers in an occupied space does not present a visibility challenge to the vehicle personnel.

## Acknowledgments

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# 1. Introduction

The mandatory phasing out of halons 1301 ( $\text{CF}_3\text{Br}$ ) and 1211 ( $\text{CF}_2\text{ClBr}$ ) for use as the primary fire-extinguishing agent aboard U.S. Army combat vehicles has led to the testing and evaluation of candidate replacement agents such as FE-36 ( $\text{C}_3\text{F}_6\text{H}_2$ ) and FM-200 ( $\text{C}_3\text{F}_7\text{H}$ ). These fluorinated propanes, though effective at extinguishing fires, are deficient in terms of increased weight and volume requirements needed for fighting the most difficult military fires. The increased amounts of replacement agent required for extinguishment, relative to halons 1301 and 1211, pose a concern with the levels of hydrogen fluoride (HF) formed as the primary decomposition product [1]. This concern has limited the acceptance of these agents for deployment in occupied spaces.

The two mechanisms by which HF concentrations can be decreased during and following fire extinguishment by FE-36 or FM-200 are by: (1) reducing the time required to extinguish the fire, thereby minimizing the time that the fluorine-containing suppressant is exposed to flame temperatures, or (2) releasing a scavenging agent in conjunction with the fire-suppressant chemical to remove HF after it is produced. A potential chemical scavenging agent of gaseous HF is ammonium polyphosphate (APP), which is a commercially available chemical (Phos-chek) with fire-retardant properties. For the tests conducted here, APP is suspended in an extinguisher as a gel and released from a nitrogen-pressurized extinguisher as a white powder. One of the drawbacks to the release of a powder agent in an occupied space is the possibility of reduced visibility.

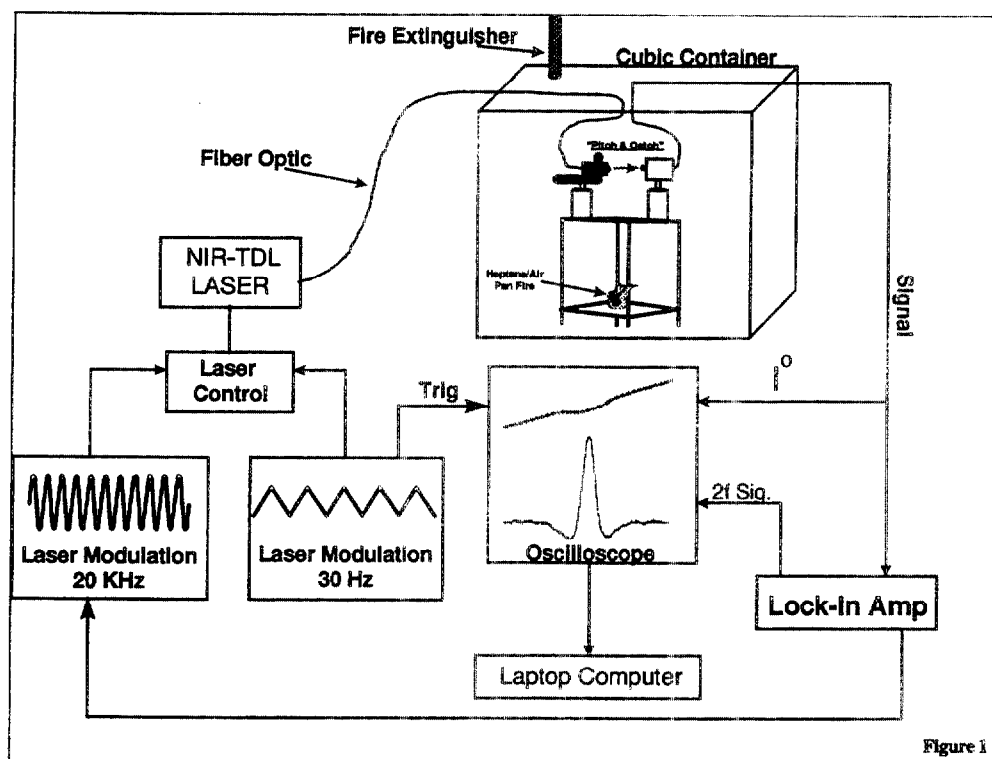
Presented here is a field study of the use of hand-held fire extinguishers containing a fluorinated propane, FE-36, and APP to extinguish liquid heptane/air pan fires. This study has focused primarily on near-infrared tunable diode laser absorption spectroscopy (NIR-TDLAS) measurements of HF concentrations produced from a series of enclosed heptane/air pan fires extinguished by FE-36 or FE-36 plus APP. A recent article from this laboratory details the use of NIR-TDLAS as the most effective diagnostic for gas-phase HF measurements [2]. A series of

extinguishment tests was conducted with FE-36 and FE-36 plus APP. By measuring the time evolution of the HF concentrations in fires extinguished with FE-36 plus APP compared to fires extinguished by FE-36 alone, the effectiveness of APP as an HF scavenging agent is demonstrated and evaluated. Separate measurements of light attenuation caused by the release of an FE-36 plus APP extinguisher inside an M1-Abrams land combat vehicle is also described and compared against an extinguisher containing  $\text{NaHCO}_3$  pressurized with  $\text{N}_2$ .

## 2. Experimental

Figure 1 shows a schematic diagram of the test facility containing the NIR-TDLAS experimental apparatus and signal-processing electronics. TDLAS has proven to be a valuable, nonintrusive, optical diagnostic for species concentrations and temperatures in combustion systems, even when the local soot level is high [3–9]. The test facility is a 1.5-m<sup>3</sup> cubic enclosure, which contains a liquid heptane pan fire (fire size approximately 30 kW). HF measurements were made using FE-36 or one of the following combinations of FE-36 plus APP as the fire suppressant: FE-36 plus 7% APP in a hand-held extinguisher pressurized to 150 psi by  $\text{N}_2$ ; FE-36 plus 7% APP in a hand-held extinguisher pressurized to 200 psi by  $\text{N}_2$ ; FE-36 plus 10% APP in a hand-held extinguisher pressurized to 250 psi by  $\text{N}_2$ , or FE-36 plus 15% APP in a hand-held extinguisher pressurized to 300 psi by  $\text{N}_2$ .

The observed test protocol was as follows: a 250-ml pan filled with heptane fuel was placed underneath 0.5-m-high steel table within the cubic container. The NIR-TDLAS HF gas measurement apparatus was situated on top of the metal table, where a calibration cell was placed in the line-of-sight path between the gradient index (GRIN) lens from which the 1.3- $\mu\text{m}$  laser radiation was emitted and the InGaAs detector. The calibration cell was used to provide an HF spectrum to tune the laser and data-collection system. The calibration cell was removed prior to fire testing. A hand-held bottle-type extinguisher charged with 770 g of FE-36 plus scavenging agent (when applicable) and pressurized with  $\text{N}_2$  gas was situated on top of the cubic



**Figure 1. Schematic Diagram of Experimental HF Measurement Apparatus.**

container. The extinguisher bottle output nozzle was attached via tubing to a spray nozzle protruding into the cubic container.

The heptane pan fire was ignited using a butane electric match, and the TDLAS HF gas measurement was initiated immediately after an internal cubic container fan was turned on and the container door closed. The heptane pan fire was allowed to burn for 15 seconds, at which time the hand-held extinguisher's contents were discharged into the interior of cubic container, producing total flooding inhibitor conditions (10% by volume FE-36 concentration). A video camera mounted within the cubic container recorded the fire event during the tests. HF data collection occurred for 180 seconds (at a rate of 1 spectrum per second) after ignition of the fuel.

Following each test, the interior of the cubic container was rinsed with an aqueous solution of sodium bicarbonate to neutralize any acid residue on the interior surface and the container was

allowed to air dry. The experimental details and analysis of NIR-TDLAS HF spectra have been described previously [2] and are only summarized here.

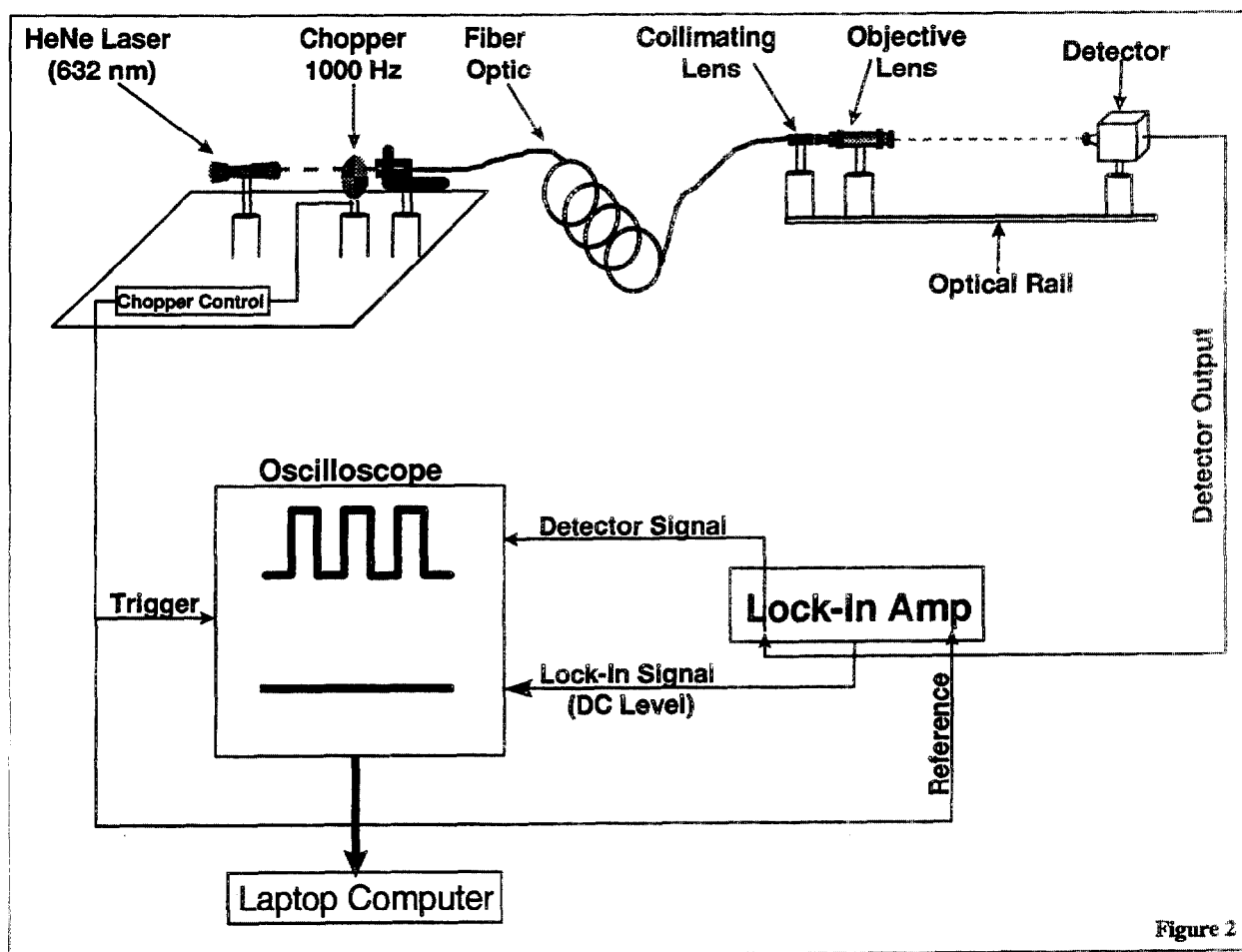
A sawtooth modulation ( $\approx 100$  Hz) supplied from a function generator (Tektronix Model FG 504) was used to rapidly scan over the desired spectral frequency range. Coincident with the 100-Hz modulation was a small-amplitude but higher frequency modulation ( $\approx 20$  kHz) taken from the sine output of a SRS Model 830 DSP lock-in amplifier. The application of the two modulations to the laser diode current facilitated wavelength modulation spectroscopy (i.e., 2f detection), which was needed for enhanced signal sensitivity [10–14]. Wavelength modulation spectroscopy is convenient for analysis because of the linear relationship that exists between the measured absorption signal and the analyte's concentration. Concentrations of HF were calibrated using a continuous flow cell containing various known mixtures of HF in  $N_2$  and measuring each mixture's unique absorption spectrum.

The emitted infrared (IR) light from the diode was launched into a fiber coupled to the laser housing. Fiber-optic patch cables were used to deliver IR light into the test facility. The fiber was terminated by a GRIN lens (Sentech Systems, Inc.), which collimated the laser radiation. The GRIN lens-tipped fiber was placed into a “pitch and catch” arrangement, which directed the IR radiation over a 14-cm open path to a InGaAs detector (Epitaxx Model ETX 1000 T). The signal observed at the detector was directed to a phase-sensitive lock-in amplifier (Stanford Research Systems [SRS] Model 830), which acquired the 2f absorption signal. The 2f output signal from the SRS lock-in amplifier was then sent to a digital oscilloscope (LeCroy Model 9654). Resulting spectra were acquired every second for a 3-minute time period and then sent to a Gateway 2000 laptop computer for storage and analysis. For experiments described here, the P(2) transition of the first vibrational overtone of HF was monitored at  $7,665\text{ cm}^{-1}$  [5].

Figure 2 presents a schematic representation of the experimental apparatus used for attenuation testing. The apparatus consists of a HeNe laser (Oriel Model 6611 output 632 nm), an Optical Chopper (SRS Model SR540), and a fiber-optic light coupler (Newport Optics Model

F-916T). The experimental equipment was mounted to a 46-cm × 46-cm optical breadboard, which was placed outside the crew compartment on top of the vehicle. A separate 41-cm optical rail supported the fiber collimating/projection optics and a 15-mm<sup>2</sup> (active element) photodetector (Centro Vision Inc. OSD5-5T, 350–1,100 nm). The optical rail was placed inside the vehicle in the driver's seat. Not shown is a plastic enclosure that was placed over the fiber collimating/projection optics to minimize the optic devices from exposure to the powder agent. An 18-m BNC cable and extension cord provided remote signal communications and power to the chopper and laser, while another 18-m BNC cable transported the detector signal out to the detection electronics. The tests consisted of an occupant releasing a Kidde 2.75-lb hand-held fire extinguisher, containing either FE-36 plus APP or NaHCO<sub>3</sub> plus N<sub>2</sub> at the personnel heater, while inside the vehicle, and all outside hatches were closed. With the hatch doors closed, the vehicle's nuclear and biological containment (NBC) system was operated to create a positive pressure inside the crew compartment.

Light attenuation is determined by monitoring the percent transmission of HeNe laser radiation that is incident on the detector as powdered chemical agent is dispersed over the measurement region. Thus, if 100% transmission occurs, all of the light that exits the laser source is incident upon the detector and no attenuation occurs. The laser radiation is passed through an optical chopper, which modulates the laser radiation at ≈1,000 Hz. After passing through the chopper, the laser radiation is launched into an 8-m-long visible fiber-optic cable (3M multimode 1,000-mm-diameter core glass-substrate optical fiber). Light passes out the opposite end of the fiber, where it is terminated into an 11-mm SMA fiber ferrule (Oriel Model 77670). The terminated fiber is inserted into a glass-collimating beam probe (Oriel Model 77645). The collimated light exits the beam probe and is directed into a visible Nikon objective lens, which apertures the beam to approximately 5–6 mm and directs it over a 0.4-m free space, where the laser radiation is incident upon the 15-mm<sup>2</sup> photodetector. The free space through which the laser radiation passes is the region in which the powdered agent, if present, attenuates the laser radiation. The light intensity reaching the detector is converted to a voltage that is then measured using a lock-in amplifier referenced to the chopper frequency. A direct-current (DC)



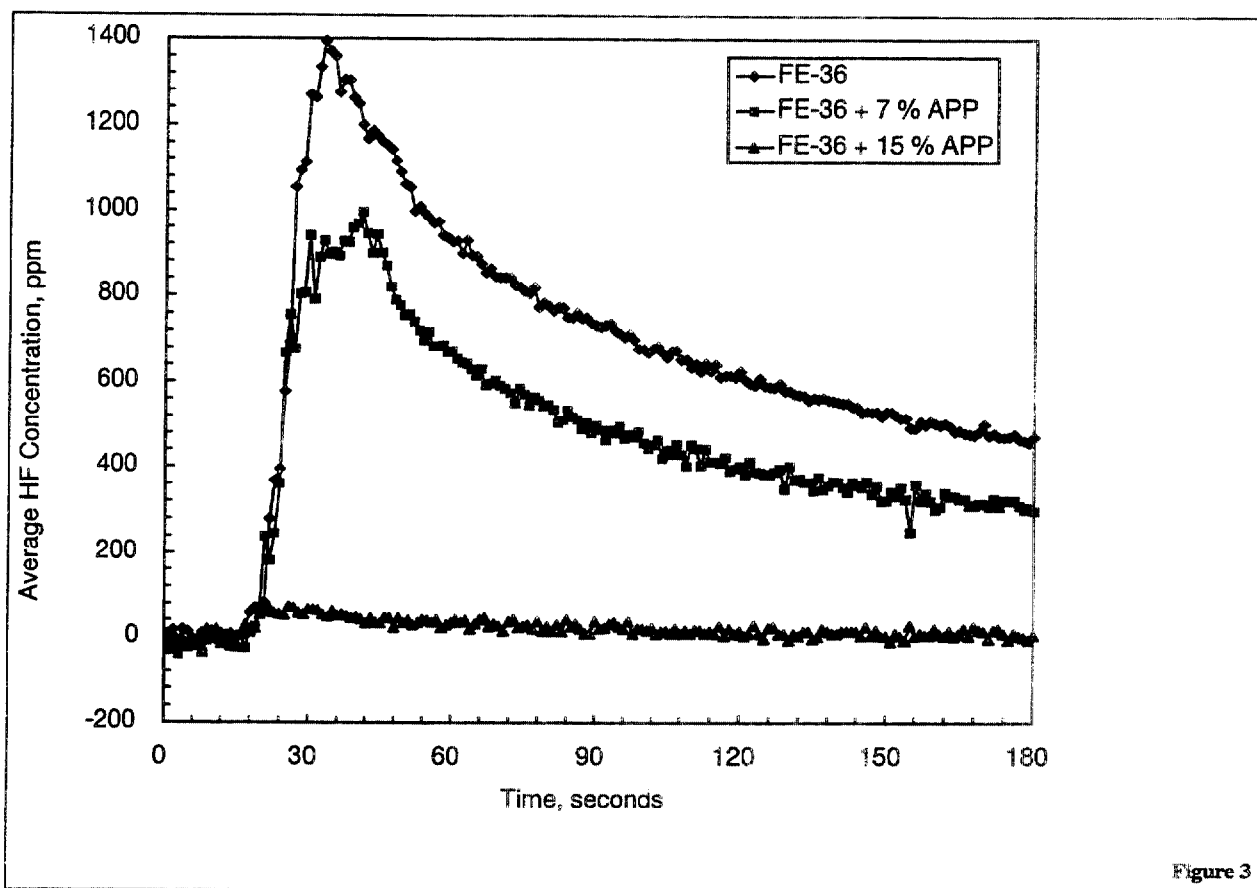
**Figure 2. Schematic Diagram of Light-Attenuation Measurement Apparatus.**

voltage (0–5-V full scale), proportional to the detector signal, is output to the digital oscilloscope (LeCroy Model 9654), which is also triggered by the optical chopper, and is processed and recorded using a Micron Trek 233-MHz laptop computer at a rate of 2 Hz.

### 3. Results

Figure 3 presents average HF concentration profiles from extinguishment tests with FE-36, FE-36 plus 7% APP, and FE-36 plus 15% APP. For the FE-36 plus 7% APP and FE-36 plus 15% APP tests, three individual profiles for each extinguisher concentration were used to



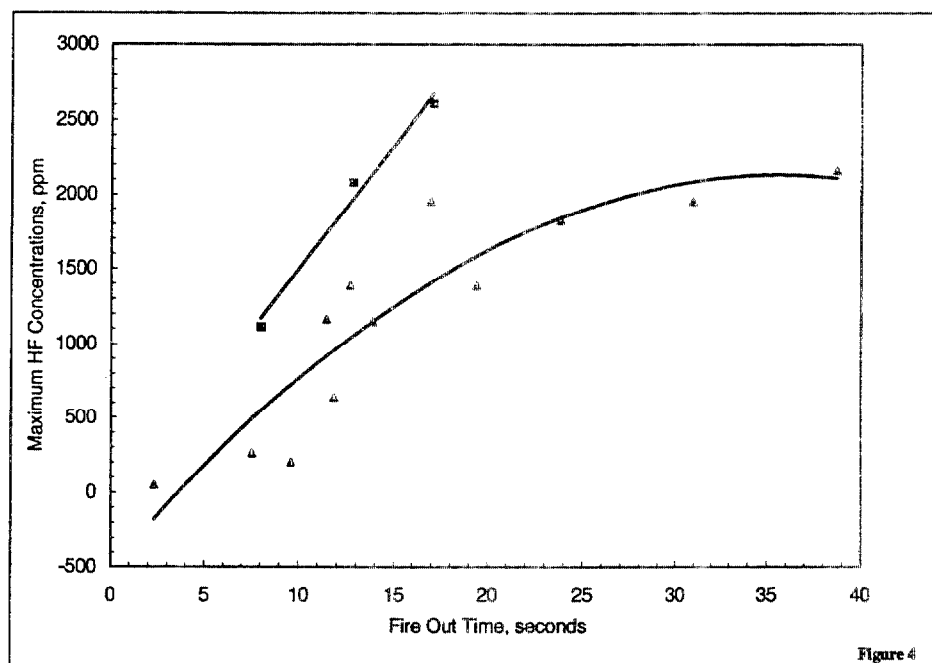


**Figure 3. Average HF Concentration Profiles vs. Measurement Time. The Diamond Symbols (◆) Are HF Concentrations From Fires Extinguished by FE-36 Only, the Square Symbols (■) Are HF Concentrations From Fires Extinguished by FE-36 Plus 7% APP, and the Triangle Symbols (▲) Are HF Concentrations From Fires Extinguished by FE-36 Plus 15% APP.**

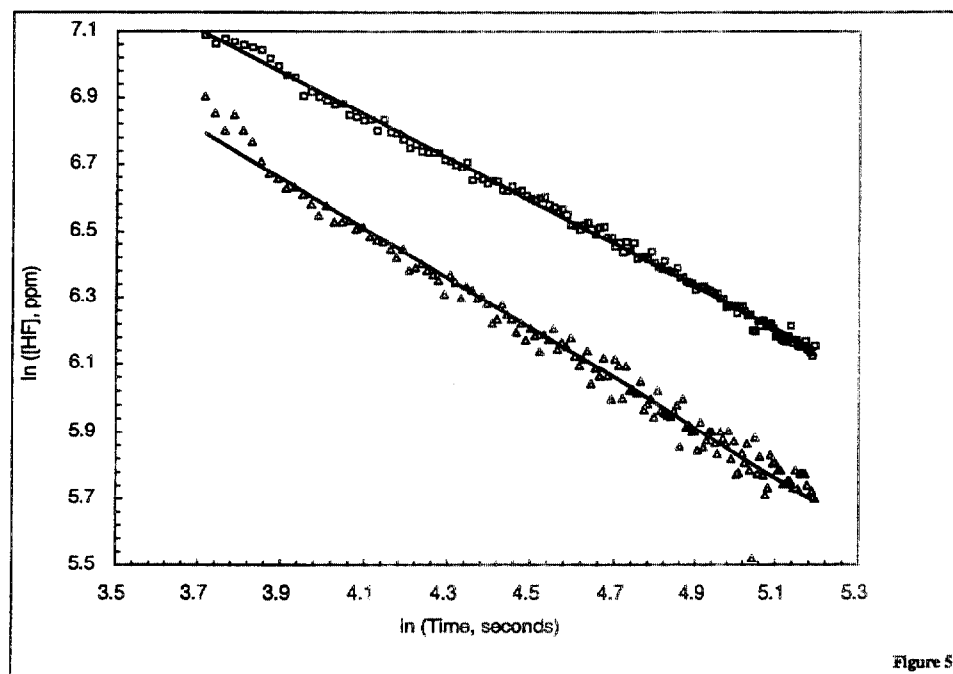
construct each test's average profile. For the FE-36 only test, data from five different profiles (collected over the entire testing period) compose the average profile. Statistically, the 1- $\sigma$  variances for the profiles were 41, 42, and 31% for FE-36 only, FE-36 plus 7% APP, and FE-36 plus 15% APP. The results indicate that, for the fires extinguished with FE-36 only, the average peak HF level was 1,394 ppm. For fires extinguished using FE-36 plus 7% APP, the peak HF concentration was 996 ppm. For fires extinguished using 15% APP, the peak HF concentration was approximately 73 ppm. The variances in the peak values were 45, 36, and 18%, respectively.

Measurement of HF gas concentration vs. time provides a monitor of the fire history and of the effectiveness of any HF scavenging agent used. That is, the time from fire-suppressant release until the maximum HF concentration occurs is a measure of the time required for fire extinguishment (also verified visually using a VHS video recorder), while the rate at which the HF concentration decreases following extinguishment provides a measure of effectiveness of scavenging agent (when used), or a measure of the rate at which HF gas reacts with the walls of the enclosure. Reduction in fire “out” times reduces HF levels by reducing the time the fluorinated agent is exposed to flame temperatures. Figure 3 shows that, for the fires extinguished by FE-36, FE-36 plus 7% APP, and FE-36 plus 15% APP, the average fire out times are 18, 20, and 12 seconds, respectively. Statistically, the difference in fire out times between fires extinguished by FE-36 and FE-36 plus 7% APP are insignificant with a 1- $\sigma$  error of 22%. For fires extinguished by FE-36 plus 15% APP, the fire out times were reduced by approximately 35%. The decrease in peak HF levels shown in Figure 3 for fires extinguished with FE-36 and fires extinguished using FE-36 plus 15% APP (approximately a factor of 10) is partially attributable to the fire-suppression properties of APP (reflected by the shorter fire out time).

Figure 4 presents the peak HF levels vs. fire out times for all fires extinguished by FE-36 and with all combinations FE-36 plus APP. The FE-36 only data illustrate that, as the fire out times increase, there is a monotonic increase in the amount of HF present in the system. When the APP is added to the extinguisher, the amount of HF measured is always less than that measured in fires extinguished using only FE-36. In real fires, reduction of fire out times is difficult to control, and, if the time duration is extended, some measures must be taken to control the HF levels. In order to select the proper concentrations of APP and operating conditions, the time rate of change of HF concentration must be measured. The rate of change in HF concentration can be compared between extinguishers using data from Figure 3, starting at the maximum HF concentration time ( $t_0$ ) and plotting the natural logarithm of the HF concentration vs. the natural logarithm of the elapsed time from the HF maximum, as seen in Figure 5. Note: because the HF concentrations for tests with FE-36 plus 15% APP were well below the target's



**Figure 4. Peak HF Concentrations vs. Fire Out Times for Fires Extinguished by FE-36 (■) and FE-36 Plus APP (▲).**

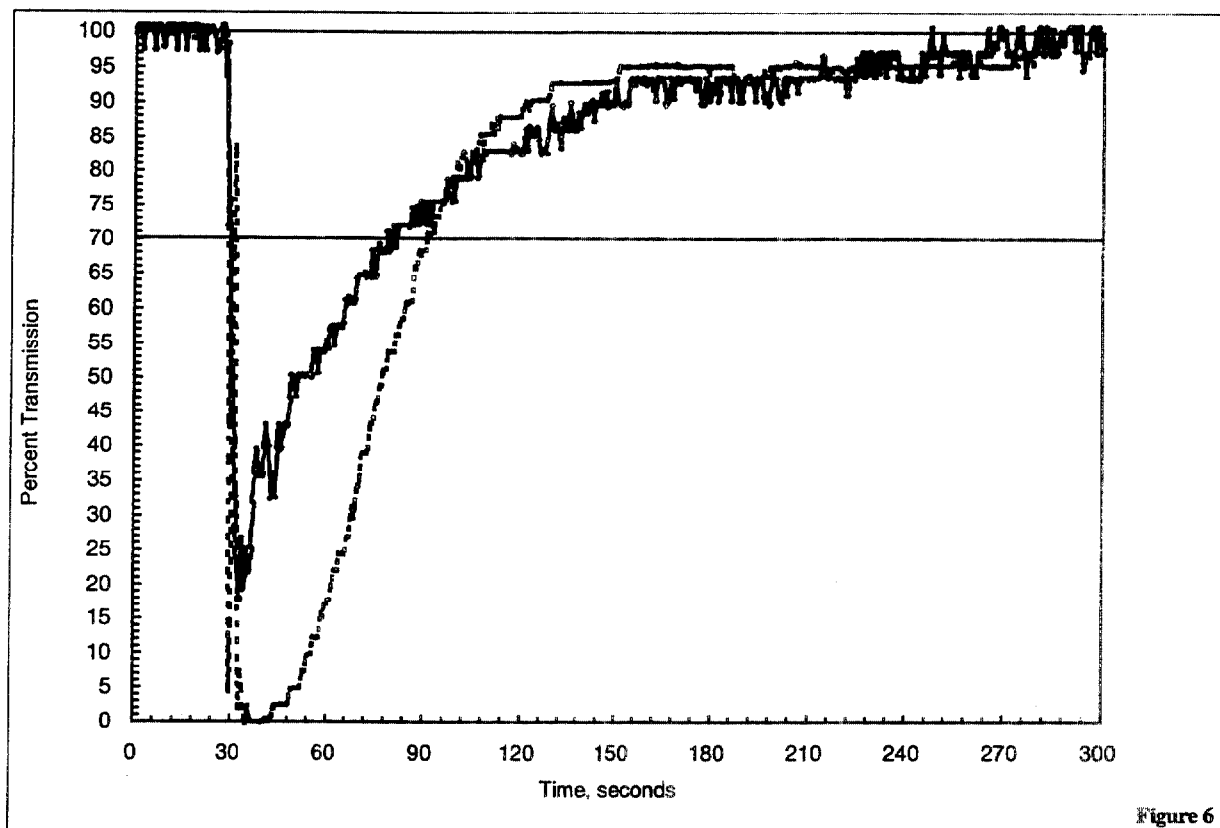


**Figure 5. Rate Plot of  $\ln$  HF Concentrations vs.  $\ln$  Time for Fires Extinguished by FE-36 (□) and FE-36 Plus 7% APP (Δ). The Straight Solid Lines Represent Linear Regression Analysis of the Data.**

toxic exposure critical concentration of 500 ppm, as well as the fact that the discharged APP concentration probably exceeded acceptable respiratory exposure levels, no further analysis of the data was warranted [15]. With Figure 5 on a ln-ln scale, the dissipation of HF vs. time is a second-order decay process. Linear regression analysis of the data in Figure 5 indicates that the FE-36 data have a slope of  $-0.645$  ppm/sec ( $R^2 = 0.991$ ), while the FE-36 plus 7% APP data have a slope of  $-0.746$  ppm/s ( $R^2 = 0.980$ ). The difference in slopes for the two tests is approximately 14% greater for the fires extinguished by FE-36 plus 7% APP, and, thus, faster decreases in HF concentrations should be expected for fires extinguished by this hybrid extinguisher.

Regardless of how fast HF is reduced in a given system, from a more practical standpoint, the amount of HF in the system over a period of time (i.e., time-weighted average) must be determined to address toxicity concerns. Again, using data from Figure 3, the time-weighted average doses of HF, calculated from the extinguisher release at  $t = 15$  seconds until  $t = 180$  seconds, were 712, 492, and 51 ppm for fires extinguished by FE-36, FE-36 plus 7% APP, and FE-36 plus 15% APP, respectively. These values imply that, for the tests with FE-36 only, the HF dose is toxic, while the tests with FE-36 plus 7% APP and 15% APP achieve minimum to very acceptable HF reductions, respectively.

Finally, Figure 6 presents the percent transmission profiles measured during and following the release of the FE-36 plus APP and the  $\text{NaHCO}_3$  plus  $\text{N}_2$  extinguishers inside the M1-Abrams land combat vehicle. The profiles indicate that the maximum light attenuations were 0% transmission for the  $\text{NaHCO}_3$  plus  $\text{N}_2$  extinguisher and 18.04% transmission for the FE-36 plus APP extinguisher. Empirical correlations using a VHS video (taken inside the vehicle during the extinguisher's release) indicate that clear visibility to the human eye correlates to an attenuation level of approximately 70% transmission. Thus, all percent transmission levels recorded below 70% correspond to an obscured field of view at a distance of 30.48 cm, while the FE-36 plus APP extinguisher experiences less than 70% transmission for 49 seconds. The more rapid return to visibility (i.e.,  $\geq 70\%$  transmission) using the FE-36 plus APP extinguisher is attributed to the



**Figure 6. Percent Transmission Profiles Collected From Measurement of Light Attenuation, While an Extinguisher Containing Either FE-36 Plus APP (Solid Line) or FE-36 Plus NaHCO<sub>3</sub> (Dashed Line) Was Released Inside an M1-Abrams Combat Vehicle. The Horizontal Line Drawn Across the Graph Is the 70% Transmission Level, Which Corresponds to Clear Visibility by the Human Eye.**

fact that the powder is not released in a dry state, but, rather, it is “wet” and thus falls faster to the vehicle floor.

## 4. Conclusions

NIR-TDLAS has been demonstrated to measure HF in a practical field application. The results presented here indicate that HF concentrations produced from fires extinguished by FE-36 plus APP are being reduced in the cubic test container and that the presence of APP accelerates

this reduction. Thus, the combination of APP in an extinguisher containing FE-36 appears to reduce HF levels. Visibility reduction during extinguisher deployment was measured inside an actual combat vehicle. The HF results should be met with caution since HF and APP threshold exposure limits inside vehicles have not yet been established. From an experimental standpoint, more tests should probably be conducted to improve statistical deviations, as well as post-testing analysis of the reacted APP, to understand how APP reacts with HF. Future tests will attempt to meet these concerns in order to develop a chemical kinetic mechanism for post-fire HF activity.

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13. ABSTRACT (Maximum 200 words)  Tunable diode laser absorption spectroscopy (TDLAS) is used to measure the time evolution of hydrogen fluoride (HF) concentrations produced from a series of enclosed heptane/air pan fires extinguished by FE-36 or FE-36 plus ammonium polyphosphate (APP). Recent advances in room-temperature fiber-coupled near-infrared (NIR) diode lasers provide isolation of the laser and signal-processing electronics from the hostile sampling site. For the fires studied, the change in HF gas concentration with time is dependent upon the fire-fighting chemical used to extinguish the fire. The presence of APP is observed to accelerate the dissipation of HF from the fire enclosure. Visible attenuation spectroscopy is also used to measure the amount of light attenuation (obscuration) that occurs as a hand-held fire extinguisher containing powder fire-fighting agent is released in the crew space of a M1-Abrams land combat vehicle. Obscuration tests demonstrate that release of APP from extinguishers in an occupied space does not present a visibility challenge to the vehicle personnel.				
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